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Effect of calcium gluconate, calcium lactate, and urea on the kinetics of self-healing in mortars

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HIGHLIGHTS

- Crack impregnation with calcium lactate increases the self-healing kinetics.
- Crack impregnation with calcium gluconate increases the self-healing kinetics.
- Impregnation increases the availability of calcium and carbonate ions.
- Thick and compact layers of calcite and ettringite in large cracks (>150 μm).

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ABSTRACT

Bacteria-based healing of cracks remaining in self-healed concrete is under development to improve durability by allowing the healing of large cracks. The objective of this research was to assess the effect of several chemical compounds (called precursors) in particular calcium gluconate, calcium lactate and urea, that could first, enhance the intensity of self-healing and second, serve as nutrients at later ages for either autogenous bacteria or bacteria subsequently added by spraying the concrete surface with a bacterial suspension. Selected solutions containing precursors were used to saturate single cracks of known geometry in mortar specimens. Airflow measurements were used to monitor the healing process and to compare the kinetics of the self-healing between precursor-saturated cracks and non-precursor-saturated cracks. The 24 h immersion of fresh cracks in mortars in calcium lactate or calcium gluconate solutions increased the self-healing kinetics for large cracks. This could increase the availability of calcium ions and carbonate ions, which are the main reagents for the formation of healing products. The higher initial availability of these reagents is confirmed by the rapid decrease of the apparent opening during the first month. The internal crack surfaces were covered with a thick, compact layer of healing products, mainly composed of calcite and ettringite.

1. Introduction

Concrete structures can be exposed to many types of harsh environments containing potentially aggressive agents (ions, gas, liquids, and humidity). Cracks are one of the most important parameters in concrete durability because they provide preferential paths for the penetration of these aggressive agents. Under particular temperature and humidity conditions, natural self-healing can almost completely seal small cracks (<150 μm) [1]. During natural self-healing, calcium carbonate and other healing products form inside cracks as a result of water and CO_2 interaction with the cement matrix [1–3]. In large cracks, however, natural self-healing is incomplete and not enough healing products are formed to completely seal such cracks [1].

Bacteria-based healing concretes are under development to improve durability by allowing for the healing of large cracks [4,5]. One of the approaches uses bacterial activity to produce calcium carbonate ($CaCO_3$) or other calcium-based minerals that progressively seal cracks [6]. The principle is called microbiologically induced calcium carbonate precipitation (MICCP). Calcium carbonate is highly compatible with concrete and offers a promising and sustainable repairing solution [7]. Bacterial strains producing calcium carbonate are alkalinophilic bacteria and thus survive from neutral to basic pH levels up to 11 [8]. These bacteria grow in specific medium and modify their environment by changing the pH, which produces new chemical products and promotes the nucleation of calcite crystals [9,10]. The various biological and

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technological approaches used for MICCP can involve a variety of precursors being added to the culture medium [11–16]. A precursor is a chemical compound providing reagents for the calcium carbonate formation, specifically, the calcium ion (Ca^{2+}) and the carbonate ion (CO_3^{2-}). Urea ($CO(NH_2)_2$) is among the most commonly used precursors [11,13–15]. When hydrolyzed by bacteria, urea provides carbonate ions and raises the pH by producing ammonium ions (NH_4^+), which are deleterious for concrete [17]. A high pH of 10 can promote calcium carbonate precipitation [18]. Other approaches involve calcium salts, such as calcium lactate ($[CH_3CH(OH)COO]_2Ca$), as potential calcium and carbonate precursors [12,16,19].

As these bacterial strains cannot develop at pH surface around 13, MICCP can only occur after a decrease in pH. However, when concrete is exposed to air for several weeks, carbonation can reduce the surface pH in a crack to a value of around 9 [1]. Carbonation is also one of the mechanisms of natural self-healing of a crack [1,8]. The objective of this research was to assess the effect of several chemical compounds (called precursors) in particularly calcium gluconate, calcium lactate and urea, that could first, enhance the intensity of self healing and second, serve as nutrients at later ages for either autogenous bacteria or bacteria subsequently added by spraying the concrete surface with a bacterial suspension.

Selected solutions containing precursors were used to saturate single cracks of known geometry in mortar specimens. Airflow measurements were used to monitor the healing process and to compare the self-healing kinetics between precursor-saturated cracks and non-precursor-saturated cracks [1]. This quantitative approach will facilitate understanding of the relative contribution of each precursor for enhancing self-healing.

2. Experimental program

2.1. Self-healing characterization

Small and large cracks were formed at 28 days in mortar samples made with a water–cement ratio (W/C) of 0.485. Small cracks were considered to have a mouth opening of less than 150 mm; large ones >150 mm. Airflow measurements were used to compute the evolution of the effective crack opening over a storage period of 6 months in a fog room at 23 °C and 100% relative humidity (R.H.). These conditions aims at simulating natural self-healing environmental conditions for open-air above-ground concrete structures. They also promote hydration and leaching and slows down carbonation compared to lower R.H. After 6 months, samples were sawn to split the crack and allow characterization of the healing products using variable pressure scanning electron microscopy (VPSEM) and energy-dispersive spectroscopy (EDS).

2.2. Mortar composition

All mortar samples had the same composition, complying with ASTM C109-C109M [20]. The W/C was 0.485 and the sand-to-cement ratio was 2.75. The cement was a Canadian general-use Portland cement (similar to CEMI and ASTM Type 1) having C_3S, C_2S, C_3A, and C_4AF, contents of 60%, 13%, 7.5% and 7.0%, respectively (Bogue composition). The Blaine fineness was 381 m^2/kg. All mortars were made with ASTM C778 compliant silica sand (Ottawa sand) having a density of 2.65 [21].

2.3. Mortar samples

Test specimens were disk-shaped mortar samples 150 mm in diameter and 50 mm thick. A central hole 55 mm in diameter was used to insert an expansive core (Fig. 1). The expansive core was used to create realistic radial cracks of controlled geometry. The detailed controlled cracking procedure was presented in a previous publication [1]. The mortar disk included an embedded steel ring to simulate a reinforcing bar balancing the internal stresses after cracking. The ring was made from a hot-rolled steel rod with an internal diameter of 95.2 mm and a sectional diameter of 4.8 mm.

The mold was filled in two layers, each being slightly vibrated for 10–15 s on a vibrating table. Immediately after casting, the fresh mortar surface was levelled and the samples were covered with a Plexiglas plate and wet burlap. After 24 h, the molds were removed and the samples stored at 23 °C and 100% R.H. until the age of 28 days.

2.4. Cracking, precursor impregnation, and airflow measurements

At the age of 28 days, the samples were subjected to cracking to produce a number of fine and large cracks with openings ranging from 95 μm to 226 μm (Table 1). The initial opening was measured with a video-microscope equipped with software tools for on-screen length measurements. The initial crack opening of each sample was computed from the average of 10 measurements made on each side (top–bottom) of a cracked mortar disk. The surface pH of samples was around 13.

Immediately after cracking, some samples were immersed for 24 h in one of the three immersion media, each containing one type of precursor. Calcium lactate (79 g/L) was used for three samples with a large crack and two samples with a small crack. Calcium gluconate (20 g/L) was used for three samples with a large crack and one sample with a small crack. Urea (10 g/L) was used for two samples with a large crack (Table 1). Six reference samples were not immersed in any solutions: the three samples with a large crack and the others with a small crack were simply kept at 23 °C and 100% R.H. for 24 h. After this initial pretreatment step,

Fig. 1. Mortar sample and expansive core.